

Analysis Chloroacetanilide and other Acetamide Herbicide Degradates in Drinking Water Using EPA Method 535 with Semi-Automated Solid Phase Extraction (EZSpe®)

Introduction

Chloroacetanilide and its congeners are degradation products of acetamide-containing herbicides. Originally, conceived as a pharmaceutical agent, its liver and kidney toxicity, led to its being phased out. However, they can leach into the environment, due to weed killer usage, posing a serious threat to human health.

EPA Method 535 outlines the procedure for extraction and analysis of these compounds in water. The extraction method outlines the use of solid phase extraction for water matrix samples employing both cartridges and disks. Consistent with other EPA 500 series methods, EPA 535 incorporates a rigid set of QC and acceptance criteria requiring precise and reproducible analytical practices. The potential for error and the variability associated with manual extractions makes the benefits of semi-automating these processes apparent.

To meet demands for a low cost method that requires less financial investment than fully automated systems, FMS developed a simple semi - automated system which is fast, inexpensive and yields high quality data.

Instrumentation

- FMS EZSpe® System
- FMS SuperVap®
- Vacuum pump
- Waters Acquity UPLC
- Waters Xevo TQD

Consumables

- FMS, Inc. 0.5 g charcoal cartridge
- FMS sodium sulfate cartridge
- Ultra pure DI water
- Ammonium acetate
- Fisher Pesticide Grade Methanol
- Restek 535 spiking standards

Procedure

- 6 samples (250 mL water each) are prepared
- Spike with various 535 standards
- Put sample bottles in place and fill rinse bottles with 15 mL 10 mM ammonium acetate in methanol
- Cartridges are installed in each of the six positions.

Stage 1:

- Vacuum is turned on
- Cartridges are conditioned with 2 x 10 mL ammonium acetate/methanol (keep wet) and 3 x 10 mL water (keep wet)
- Samples are loaded across cartridges under vacuum at 10 mL/min
- Cartridges are rinsed with 5 mL water and dried with nitrogen for 3 minutes.
- Sample bottles are automatically rinsed from the rinse bottles with 15 mL ammonium acetate/methanol.

Stage 2:

- Ammonium acetate/methanol from sample bottles is loaded across the cartridges (5 mL, then 10 mL) and the eluent is collected for analysis into Direct to LC Vial Collection Vessels

FMS SuperVap®

- Pre-heat temp: 65 °C
- Pre-heat time: 15 minutes
- Heat in Sensor mode at 65 °C under nitrogen (7-10 psi)
- Direct to LC Vial Vessel Reduce to 1 mL
- Samples are now ready for analysis

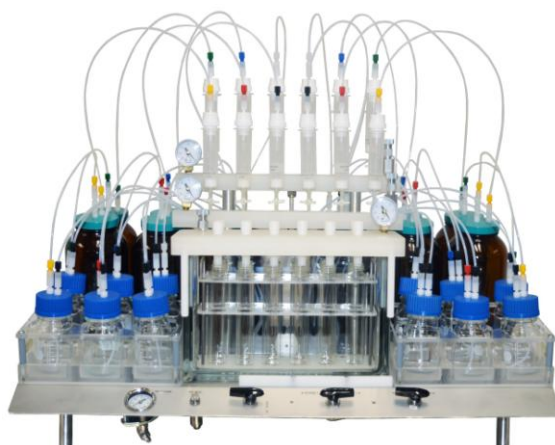


Table 1 with recoveries for a number of 535 compounds

Compound	%Recovery	Stdev
Acetochlor ESA	96.2	4.2
Acetochlor OA	83.5	8.2
Alachlor ESA	89.1	6.0
Alachlor OA	97.3	7.4
Dimethenamid ESA	102.5	3.8
Dimethenamid OA	91.2	5.3
Flufenacet ESA	102.4	5.9
Flufenacet OA	79.5	4.7
Metolachlor ESA	91.6	6.3
Metolachlor OA	84.5	3.5
Propachlor ESA	92.7	6.7
Propachlor OA	100.3	4.5

Conclusions

Reviewing the sample data shows high recoveries for a dozen acetanilide congeners, demonstrating excellent efficiency for these compounds. Samples can be taken from collection bottle to LC vial in one quick, consistent, reproducible process that will save laboratories both time and money.



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FMS EZSpe® System